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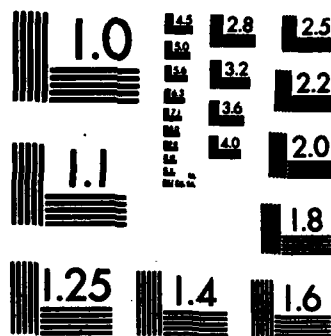
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Correlation of Electronic Charge Transfer Transitions and Electrochemical Potentials. The Bipyrzine(Tetracarbonyl)Molybdenum(O) System in Various Solvents

BY

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Correlation of Electronic Charge Transfer Transitions and Electrochemical Potentials. The Bipyrazine(Tetracarbonyl)-Molybdenum(0) System in Various Solvents

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Abstract

Using a free energy diagram, a relationship is drawn between an optical charge transfer energy and the electrochemical potentials of the donor and acceptor orbitals concerned. The charge transfer spectroscopy and electrochemical potentials of the title complex were studied in various solvents. A linear correlation, with negative slope, was observed between the difference in oxidation and reduction potentials and an MLCT transition. Using some additional solvent data, a number of useful parameters were derived in a fashion which would not be possible through consideration of either technique alone.

An electronic charge transfer transition involves excitation from a donor orbital in the ground state of a molecule, to an acceptor orbital in a Franck-Condon (non-equilibrium) excited state of the molecule. In this paper we refer specifically to an MLCT transition, from an orbital,  $\psi_M$ , mainly on the metal, to an orbital,  $\psi_L$ , primarily on the ligand. However the treatment is quite general.

An electrochemical study may, in appropriate cases, define the redox potentials of orbitals related to those above. Thus we may

observe the oxidation potential of  $\psi_M$  in the ground state of the complex  $ML_n$ , and the reduction potential for adding an electron to  $\psi_L$  to form the ground state of the reduced species  $ML_n^-$ .

Previous authors [1-5] have demonstrated qualitative relationships between optical charge transfer energies and electrochemical potentials but now we seek a more quantitative understanding in which quantities not derivable from either electronic spectroscopy or electrochemistry alone, can be obtained by a combined analysis.

In view of the solvation contributions to the parameters obtained with each technique, a combined analysis is best approached within the framework of the collection and comparison of data in a range of solvents. The analysis which follows is based upon theories developed by Born [6], Onsager [7], Kirkwood [8], Lippert [9], Marcus [10] and Meyer [11].

Use of a free energy cycle (Fig.1) permits one to relate the various thermodynamic quantities (as defined in the legend to Fig.1), measurable in the two experimental regimes. Thus the following equalities may be written:-

$$E_{op} = \Delta E_s + \chi_i + \chi_o \quad (1)$$

$$\Delta E_s = \Delta E_g + \Delta(sol) \quad (2)$$

$$\begin{aligned} \Delta E_g &= nF\Delta E(\text{redox}) + \Delta\Delta G_s + Q \\ &= (nF\Delta E'(\text{redox}) + x) + \Delta\Delta G_s + Q \text{ (see text below)} \end{aligned} \quad (3)$$

and hence:-

$$E_{op} = [X_i + nF\Delta E(\text{redox}) + \Delta\Delta G_s + Q] + X_o + \Delta(\text{sol}) \quad (4)$$

where  $X_o + \Delta(\text{sol})$  is the total change in solvation free energy as earlier defined in [10b] eqn.(13), and [9] eqn.B2a, and  $X_i$  and  $X_o$  are the inner sphere (vibrational) and outer-sphere (solvent, but also vibrational in nature) contributions to the reorganisation energy of the transition, respectively. Note that as a consequence of the Franck-Condon nature of the excited state, the transition energy  $E_{op}$  is a free energy in that the entropies of the ground and non-equilibrated excited state are the same [10b].

Equation (4), in which the square bracketed function is solvent independent for a given molecule, provides the important correlation between the two techniques.

The complex  $\text{Mo(CO)}_4\text{bpz}$  (bpz = bipyrazine) [12] displays a strong band, inter alia, attributed to  $d(\text{Mo(0)}) \rightarrow \pi^*(\text{bpz})$  MLCT, whose energy,  $E_{op}$ , varies from about 16,000 to 20,000  $\text{cm}^{-1}$  depending upon the solvent environment and shifting to higher energy with increasing solvent polarity, (Table 1) (cf the solvatochromism of the bipyridine analog [13]). Electrochemical data for a range of solvents are also shown in Table 1.

The reduction potential for the electrochemically reversible  $\text{Mo(CO)}_4\text{bpz}/\text{Mo(CO)}_4\text{bpz}^-$  couple involves addition of an electron to  $\psi_L$  ( $\pi^*$  bpz) forming the radical  $\text{bpz}^-$  bound to  $\text{Mo(0)}$ . This couple shifts to more positive potentials with increasing polarity of the solvent, as the ground state radical anion becomes more solvent

stabilised.

Oxidation occurs at the Mo(0) d manifold but is electrochemically irreversible, due to a following chemical reaction ( $EC_i$  mechanism) [15,16]. However the slow step of the following reaction appears to be Mo-CO bond breaking which is solvent independent. Thus the true thermodynamic potential will be more positive than recorded in Table 1 by a solvent independent quantity, say  $x$  mV (probably  $100 < x < 300$ ). Thus  $\Delta E(\text{redox})$  in (4) is replaced by  $\Delta E'(\text{redox}) + x$ , where  $\Delta E'(\text{redox})$  is the experimental difference between the oxidation and reduction potentials.

A plot of  $E_{op}$  against  $\Delta E'(\text{redox})$  is linear with negative slope (correlation coefficient 0.98):-

$$E_{op} = -1.04 \Delta E'(\text{redox}) + 33,950 \quad (\text{in cm}^{-1})$$

(5)

This is an unexpected result appearing to contradict intuition in that as the potential difference between donor and acceptor orbital decreases, the optical transition energy increases. Such an inverse correlation has not been previously reported. An understanding of this phenomenon develops from further analysis below.

The expression (6) [8] allows one to approximate the solvation free energy of a species as a power series:-

$$\Delta G_s = 0.5 \sum_{n=0}^{\infty} ((n+1)Q_n/b^{2n+1}) [(1-D_s)/((n+1)D_s + n)]$$

(6)

where terms higher than  $n = 1$  are ignored and the reader is



referred to the earlier literature [6-11] for detailed discussion of the conditions under which this expression is useful.  $D_s$  is the static dielectric constant of the solvent,  $b$  is the radius of the solute,  $Q_0 = z^2 e^2$ , and  $Q_1 = \vec{\mu}^2$ , where  $\vec{\mu}$  is the dipole moment of the solute species. The term in  $n = 0$  disappears for uncharged species. The difference in solvation free energies of the uncharged and thermally equilibrated ground and excited states,  $\Delta(sol)$ , may be written using (6) as:-

$$\Delta(sol) = \Delta G_s(e) - \Delta G_s(g) = ((\vec{\mu}_e^2 - \vec{\mu}_g^2)/b^3)[(1-D_s)/(2D_s+1)] \quad (7)$$

Following earlier authors [8-11], the Franck-Condon destabilisation of the excited state, due to solvent interactions, may be written:-

$$\chi_0 = ((\vec{\mu}_e - \vec{\mu}_g)^2/b^3)[(1-D_{op})/(2D_{op}+1) - (1-D_s)/(2D_s+1)] \quad (8)$$

where in (7,8) the ground and excited state dipole moments are appropriately discriminated, and  $D_{op}$  is the optical dielectric constant of the solvent (square of the refractive index). The total solvent dependence excluding non-polar contributions (very small [10b]), (eqn.(4)) is  $\Delta E_T = \Delta(sol) + \chi_0$ ; summing (7) and (8) yields (9) (after evaluating the vector products) [9,11]:-

$$\begin{aligned} \Delta E_T = & ((\mu_e^2 + \mu_g^2 - 2\mu_e\mu_g\cos\theta)/b^3)[(1-D_{op})/(2D_{op}+1)] \\ & + 2\mu_g(\mu_e\cos\theta - \mu_g)/b^3[(1-D_s)/(2D_s+1)] \end{aligned} \quad (9)$$

The angle  $\theta$  is that between the ground and excited state

dipoles. The ground state dipole moment lies along the  $C_2$  axis with the negative end pointing towards the CO groups.

Equation (4) can now be recast in the general form:-

$$E_{op} = \chi_1 + \Delta E_g + f(1-D_{op})/(2D_{op}+1) + f'(1-D_g)/(2D_g+1) \quad (10)$$

where  $f$  and  $f'$  group the factors shown in eqn.(9).

In a forthcoming paper [15] we develop this analysis with 23 solvents. Here we shall restrict ourselves to considering six solvents of low polarity and low dielectric constant which might be expected to obey the dielectric continuum model. The optical data thereof (Table 1) obey (10) well according to (in  $\text{cm}^{-1}$ ):-

$$E_{op} = 9500((1-D_{op})/(2D_{op}+1)) - 7600((1-D_g)/(2D_g+1)) + 16,800 \quad (11)$$

with a correlation coefficient of 0.988 and a standard deviation of 65 (the lower set of six solvents shown in Table 1 were used), and where the constant term,  $16,800 \text{ cm}^{-1}$  is associated with  $\chi_1 + \Delta E_g$ . However the data yield a family of solutions which do not differ greatly in their correlation coefficients or standard deviations. Indeed the solution  $f = 0$ ,  $f' = -7110$ , and  $\chi_1 + \Delta E_g = 15,110 \text{ cm}^{-1}$  is not unreasonable (correlation coefficient 0.97, standard deviation 169). This latter solution is, however, inconsistent with the expressions in (9). The solution in (11) is statistically the best, but does not differ significantly from many other solutions in which  $f$  lies between zero and  $9500 \text{ cm}^{-1}$ . However  $f'$  is generally found in the range  $-(7000 - 7700) \text{ cm}^{-1}$  and the constant ranges from about  $15,000 - 17,000 \text{ cm}^{-1}$ . At this stage

of study we note that the approach seems justified but that higher quality and more extensive data are necessary.

The negative value for  $f'$  requires, considering equations (8-11), that, in this system, the dipole moment changes direction by  $180^\circ$  in the excited state relative to the ground state [2,15]. This provides an understanding of the Franck-Condon destabilisation of the excited state by polar solvents, and hence of the negative correlation shown in (5).

For the purpose of indicating how these data may be utilised, we choose a median value of the constant of  $16,000 \text{ cm}^{-1}$ .

Note that in the gas phase,  $\Delta\Delta G_g = 0$ , therefore  $X_1 + nF\Delta E'(\text{redox}) + x + Q = 16,000 \text{ cm}^{-1}$ . Further, use of eqn.(5) provides the value for  $nF\Delta E'(\text{redox})$  in the gas phase by insertion of  $E_{op} = 16,000 \text{ cm}^{-1}$ . Knowing  $\Delta E'(\text{redox})$  allows derivation of  $X_1 + x + Q = -1260 \text{ cm}^{-1}$ . This then leads to the evaluation of  $\Delta\Delta G_g$  (eqn.3). Use of eqn(4) leads to evaluation of  $\Delta(\text{sol}) + X_0$ . These data are also collected in Table 1.

The sum  $(X_1 + x)$  is necessarily positive but  $Q$ , using an appropriate free energy cycle, is estimated to be near  $-0.5V$  [15]. The 'gas phase' optical energy is close to that observed in pentane, as might be anticipated. The procedure provides an interesting set of parameters whose values seems eminently reasonable. Further development on this and related systems should provide the impetus to link the study of electrochemistry and optical spectroscopy and to seek evaluation of other useful parameters, such as the self-exchange energy, which may be derivable from a free energy cycle involving  $Q$ . The merits and boundary conditions inherent in this analysis will be discussed in

more detail in a future publication [15]. We also currently seek emission data which should further define some of the parameters derived.

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Table 1

Electrochemical and Spectroscopic Data<sup>a</sup>

|                   | $E(\text{Mo(I)}/\text{Solvent Mo(0)})^b$ | $E(\text{bpz})/\text{bpz}^-$ | $\Delta E'(\text{redox})$ | $E_{\text{op}}$  | $\chi_o + \Delta(\text{sol})$ | $\Delta \Delta G_s$ |
|-------------------|--|------------------------------|---------------------------|------------------|-------------------------------|---------------------|
|                   | eV                                       | eV                           | eV                        | $\text{cm}^{-1}$ | $\text{cm}^{-1}$              | $\text{cm}^{-1}$    |
| DMF               | 0.26                                     | -1.42                        | 1.68                      | (13,550)         | 19,650                        | 3650                |
| PC                | 0.32                                     | -1.41                        | 1.73                      | (14,000)         | 19,550                        | 3550                |
| MeCN              | 0.28                                     | -1.45                        | 1.73                      | (14,000)         | 19,450                        | 3450                |
| PY                | 0.26                                     | -1.52                        | 1.78                      | (14,350)         | 18,900                        | 2900                |
| THF               | 0.30                                     | -1.52                        | 1.82                      | (14,700)         | 18,650                        | 2650                |
| DCE               | 0.34                                     | -1.53                        | 1.87                      | (15,100)         | 18,150                        | 2150                |
| DCM               |  |                              | 1.90 <sup>c</sup>         | (15,350)         | 17,990                        | 1990                |
| Et <sub>2</sub> O |  |                              | 1.93 <sup>c</sup>         | (15,530)         | 17,790                        | 1790                |
| CHCl <sub>3</sub> |  |                              | 1.98 <sup>c</sup>         | (15,950)         | 17,360                        | 1360                |
| TCM               |  |                              | 2.07 <sup>c</sup>         | (16,670)         | 16,610                        | 610                 |
| Pent              |  |                              | 2.09 <sup>c</sup>         | (16,870)         | 16,400                        | 400                 |
| 'Gas'             |  |                              | 2.14 <sup>c</sup>         | (17,260)         | 16,000                        | 0                   |

a) All electrochemical potentials referenced against ferrocene as internal referant ( $\text{Fc}^+/\text{Fc}$  is at 0.16 eV vs. sce) [14]. Potentials were recorded on Pt electrodes using cyclic voltammetry at scan rates of 100, 50 and 20 mV/s. Confirmatory data were obtained using differential pulse polarography. Data are averages of several experiments. b) The oxidation process is electrochemically irreversible.  $E_p - E_{p/2}$  is comparable to that of the ferrocene couple under the same conditions. Value quoted is  $1/2(E_p + E_{p/2})$ . c) Calculated from eqn.(5) in text. NB  $\Delta E_g + \chi_i = 16000 \text{ cm}^{-1}$ ,  $\chi_i + x + Q = -1260 \text{ cm}^{-1}$ .

DMF = dimethylformamide; PC = propylene carbonate; PY = pyridine; THF  
= tetrahydrofuran; DCE = 1,2-dichloroethane; DCM = dichloromethane;  
TCM = tetrachloromethane; Pent = pentane. 'Gas' is extrapolation  
using eqn.(11).

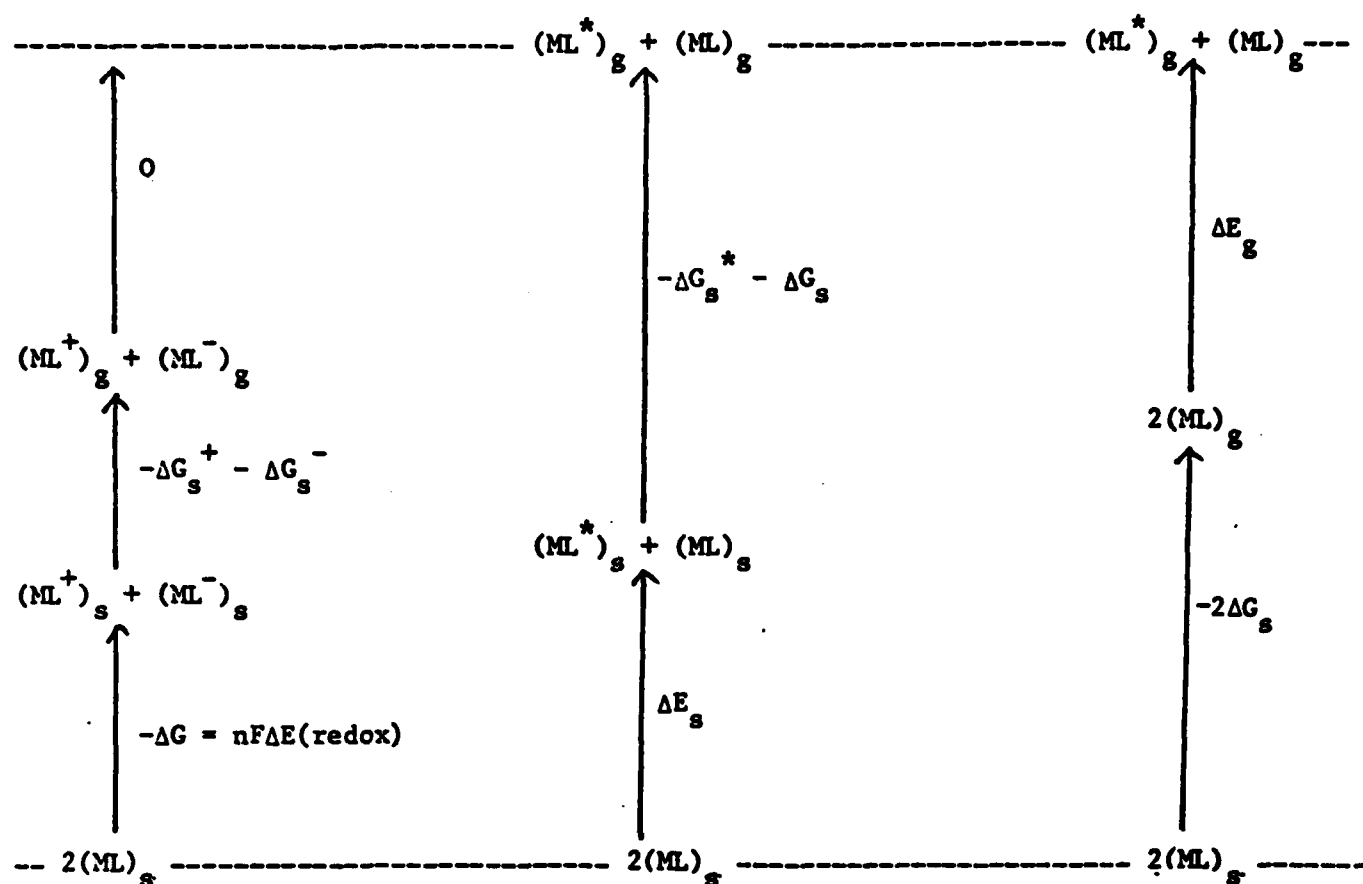


Fig.1 Free energy diagram relating spectroscopic and electrochemical quantities.

(ML) is the neutral species, and  $(ML^*)$ ,  $(ML^+)$  and  $(ML^-)$  are the equilibrated excited, mono-positive and mono-negative species respectively. Similarly  $\Delta G_s$ ,  $\Delta G_s^*$ ,  $\Delta G_s^+$  and  $\Delta G_s^-$  are the solvation free energies for these respective species being defined as the reversible work to transfer these solutes from the gas phase to a solvent.  $\Delta E(\text{redox})$  is defined as the oxidation potential minus the reduction potential of the neutral species.  $\Delta E_g$  is the gas phase excitation energy to the equilibrated excited state.  $\Delta E_s$  is the solvent phase excitation energy to the equilibrated excited state. The quantity  $Q$  is the resonance energy involved in transferring an electron from  $(ML^-)_g$  to  $(ML^+)_g$  yielding  $(ML)_g + (ML^*)_g$ . Note that in the text  $\Delta \Delta G_s = 2\Delta G_s - \Delta G_s^+ - \Delta G_s^-$  and  $\Delta(\text{sol}) = \Delta G_s^* - \Delta G_s$ .



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